DOI 10.36622/VSTU.2021.52.4.007
UDC 666.9.03

G. S. Slavcheva ¹, E. A. Britvina ², M. A. Shvedova ³

FRESH PROPERTIES AND MIX DESIGN FOR 3D-PRINTABLE DECORATIVE CONCRETE *

Voronezh State Technical University ^{1,2,3}
Russia, Voronezh

¹ *D. Sc. in Engineering, Prof. of the Dept. of Technology of Building Materials, Products and Construction, e-mail: gslavcheva@yandex.ru*

² *PhD student of the Dept. of Technology of Building Materials, Products and Construction, e-mail: esolovieva@vgasu.vrn.ru*

³ *Engineer of the Department of chemistry and chemical technology of materials, e-mail: marishwedowa@mail.ru*

Statement of the problem. This paper presents the rheological properties of 3D-printable decorative concrete. The effects of mix proportion on its plasticity and shape stability are presented together. It has been established that kind of cement changes plasticity of fresh mixtures and its resistance to load during the printing.

Results. The fresh mixtures of 3D-printable decorative concrete with effective mix design had plastic yield value $K_i \cong 1.0 - 2.2$ kPa, structural strength $\sigma_0 = 1.5 - 4.5$ kPa, value of plastic deformations $\Delta_{pl} = 0.03 - 0.07$ mm/mm. That is defined the ability of these mixtures to plastically deform without structure destruction and hold its shape, resist the deformation under compressions load during multi-layer casting.

Conclusions. Shape stability of 3D-printable mixture can be changed by 2—3 times by using cement with efficient ranging of a particle size. The plasticity and shape stability of fresh mixtures can be regulated with usage of viscosity modifiers, the type of which depends on the type of cement.

Keywords: additive manufacturing, 3D-printable decorative concrete, rheological properties.

Introduction. The decorative (architectural) concretes are increasingly used for molding products of relief surfaces, sculptures, facing slabs, making floors, outdoor furniture, paving slabs, produced with traditional concrete technology. Architectural concrete is considered a special one [4], which stresses the color and texture of the finished surface [3, 6, 12, 13]. To decorate the appearance of concrete, the white cement, and the pigments guarantee the added surface value. The white cement modifies the lightness, and the addition of small amounts of pigment provides saturation and hue properties.

3D Concrete Printing (3DCP), also known as additive manufacturing, is an emerging technology that allows structures to be built additively and thereby eliminate the use of formwork.

© Slavcheva G. S., Britvina E. A., Shvedova M. A., 2021

*This research was supported by Project # 3.1.1.2 within the 2021 Plan of Fundamental Research of the Russian Academy of Architecture and Civil Engineering and the Ministry of Civil Engineering and Public Utilities of the Russian Federation. The experimental studies have been carried out using the facilities of the Collective Research Center named after Professor Yu.M. Borisov, Voronezh State Technical University, which is partly supported by the Ministry of Science and Education of the Russian Federation, Project No 075-15-2021-662.

Building without formwork has a lot of advantages such as printing the freeform constructions, savings in cost, time, and labor [5, 11]. Among them, the main advantage of 3DCP for the architectural expressiveness of buildings is the freeform of 3D-printed structures. Expansion of the range of products from decorative concrete is associated with the possibility of 3DCP printing the freeform constructions without formwork. The development of surface finish possibilities of 3DCP makes it an attractive material to be incorporated in modern architecture. However, the world practice of 3D-build printing shows that the printed structures need finishing because 3D-printable cement-based materials do not have a decorative effect.

The kind of cement determines not only the appearance of the concrete but also its rheological, physical, mechanical, and durability properties. Different kinds and proportions of additives allow developing 3D-printable cementitious mixtures with a wide range of properties either in the fresh or the hardened states [7, 14—19]. Therefore, rheological properties such as plasticity and shape stability are significant properties for all printable materials [8—10, 20, 21, 23].

The author's theoretical approach to identifying the main factors of stability of the 3D printable materials is based on the analysis of the 3D printable disperse systems behaviour in dynamic (during pumping and extrusion) and static (during multi-layer casting) conditions of 3D-printing according to point of view of classic structural rheology of disperse systems [2]. In [1] we justified basic means of the rheological behavioural management of 3D-printable mixtures considered as a system “solid phase — liquid phase”.

In connection with the solid phase, they include its concentration, size of particles and their morphology, chemical-mineralogical composition, physicochemical characteristics of a particle surface. In connection with the liquid phase, they include its ionic composition, viscosity, density. Thus the main technological means of regulating the rheological behaviour of mixtures include three grades of factors. The first one is the water/cement ratio as a factor defining the concentration of solid phase in the system. The second one is concentration and kind of additives as a factor defining the properties of a liquid phase. The third one is the concentration, type, and granulometry of cement, fillers, and aggregates.

This article is dedicated to the study of the influence of the kind of cement and viscosity modifying additive on the rheological behaviour of 3D printable decorative concrete.

1. Materials and methods. For types of 3D-printable decorative mixtures were studied (Table 1). Table 2 illustrates the chemical composition and particle size of the mixture's initial components. Kinds of cement and viscosity modifying additive were assumed as the main factors for the change of rheological behaviour of the 3D-printable mixture. The consistency of mixtures was assumed as a fixed factor for the experiments. To evaluate the extrudability and firm stability

of fresh mixtures, the different kinds of squeezing tests were based on the methodology developed in the works. Cylindrical samples of fresh mixture with radius R equal to their height $h_0 = 25$ mm were used for the implementation of the experiment. For the squeezing test, the sample was put between two smooth plates whose diameter corresponded to the size of the sample and was loaded into the testing system “INSTRON Sates 1500 HDS”. The test was conducted on a fresh sample for all the compositions of the mixture directly following their molding.

Table 1

Mix composition and specimen identification

Specimen ID	Cement kind	Cement / limestone filler ratio	Plasticiser / mass cement (%)	Viscosity modifying additive		Fiber / mass cement (%)	W/C
				kind	mass cement (%)		
C1-LF-P-M-F	CEM I 42.5 R (grey)	1 : 1	1.2	metakaolin	2	0.5	0.20
C1-LF-P-PP-XG-F	CEM I 42.5 R (grey)	1 : 1	1.2	xanthan gum + $K_4P_2O_7$	0.2 + 0.2	0.5	0.19
C2-LF-P-M-F	CEM I 52.5 R	1 : 1	1.2	metakaolin	2	0.5	0.20
C2-LF-P-PP-XG-F	CEM I 52.5 R	1 : 1	1.2	xanthan gum + $K_4P_2O_7$	0.2 + 0.2	0.5	0.19

Table 2

The initial components of the 3D-printable mixtures

Initial component	Chemical composition	Particle size	
		ω particle, %	d , nm
Ordinary Portland cement CEM I 42.5 R (grey) (C1)	C_3S — 60.85 %, C_2S — 13.38 %, C_3A — 7.42 %, C_4AF — 11.23 %	41.9	50
		33.0	30
		19.3	12
		5.3	2
High-strength Portland cement CEM I 52.5 R (white) (C2)	C_3S — 66.07 %, C_2S — 17.48 %, C_3A — 15.42 %, C_4AF — 1.03 %	31.9	50
		23.0	30
		39.0	12
		5.6	2
The plasticizer of Sika ViscoCrete T100 (P)	Polycarboxylate ethers 100 %	—	—
Limestone filler (LF)	$CaCO_3$ ~ 95%	10	55
		10	27
		19.8	10
		14.2	2
		7.8	1
Sika Fiber	Polypropylene 100 %	$l = 12$ mm, $d = 0.022 - 0.034 \mu m$	
FUFENG®80 xanthan gum (XG)	$(C_{35}H_{49}O_{29})_n$ ~ 91 %	41	180
		35	200
		15	250
VMK-45 metakaolin (M)	Al_2O_3 — 47 %, SiO_2 — 53 %	24	1
		65	2
		7	4.9
YUCHENG commercially pure potassium pyrophosphate (PP)	$K_4P_2O_7$ — 98 %	38	0.15
		41	0.20
		16	0.25

A high compression speed test using constant plate speed $\nu = 5$ mm/s was implemented as the behaviour of the system in the process of extrusion is most adequately modeled with this speed. The curves “compression force N — displacement Δ ” obtained during the experiments were interpreted as influence curves of reduced compression load F^* from the relative change of height of the sample h_i/R . According to the results of the analysis of the received experimental curves for the studied system's values K_i , called plastic yield value by N. Roussel and C. Lanos [22], were calculated at the inflection point of the $F^*(h_i/R)$ (Fig. 1):

$$K_i = \left(\frac{h_i}{R} \right) = \frac{\sqrt{3F^*}}{2}, \quad (1)$$

where $h_i = (h_0 - \Delta)$, h_0 is the initial height of the sample, Δ is transferred in the I point of time, value R was taken as constant and equal to the radius of the sample at the beginning of the experiment.

The squeezing test was conducted with a constant strain rate $\nu = 0.5$ N/s was implemented as the behavior of the system in the process of multi-layer casting. The strain rate conforms to the average speed of load increase during multi-layer casting of building sites by industrial printers. Thus, the load on the first poured layer from gradually increasing pressure of upper layers during 3D-printing was simulated. Squeezing was conducted until the rupture of the samples, during the experiments the curves “displacement Δ — time t ”, “compression force N — displacement Δ ” were recorded. Based on the obtained experimental curves, values of the structural strength of cement pastes were calculated for the moments corresponding to the start of deformation and of cracking in the samples:

$$\sigma = \frac{P}{\pi R^2}. \quad (2)$$

Thus, the rheological properties of fresh mixtures were evaluated based on the following criteria:

- plastic yield value K_i ,
- structural strength σ_0 at the beginning of the deformation,
- plastic strength σ_{pl} was calculated at the beginning of cracking,
- the value of plastic deformations Δ_{pl} at the start of cracking was determined as a ratio of the displacement Δ of the plates in the squeezing test to the initial height of the sample h_0 .

At the same time, the temperature of the mixes was controlled during setting with a contact alcohol thermometer. The thermometer was installed in a glass test tube with vaseline oil,

fixed in the center of a sample of a freshly prepared mix. The temperature measurement was continued until the mixture temperature stabilized.

2. Experimental results and discussion. As a result of the interpretation of the squeezing test with constant plate speed, experimental curves $F^* = f(h_i/R)$ (Fig. 1, a) were obtained which correspond to the similar curves of N. Roussel [24]. Analysis of experimental curves $F^* = f(h_i/R)$ for a description of rheological behaviour of 3D-printable decorative mixtures during squeezing was conducted based on approaches of fundamental structural rheology of disperse systems [21]. Under the action of low compression stress on the first section of the curve within deformation range $\sim 0.8 < h_i/R < 1$ the structure maintains stability ("placing phase" according to N. Roussel's terminology). From the standpoint of structural rheology of disperse systems, the "placing phase" can be characterized as a viscoplastic fluid with an undisturbed structure. When the stress on the second section increases with $0.5 < h_i/R < 0.8$, the system is plastically deformed while its structure loses its stability ("perfect plastic response phase" according to N. Roussel).

This section can be correlated with a viscoplastic fluid with an intensively damaged structure in compliance with the structural rheology approach. The experimental results show the different kinds of $F^*(h/R)$ curves for all the investigated 3D-printable systems (Fig. 1, a). The systems C1-LF-P-M-F, C1-LF-P-PP-XG-F, C2-LF-P-PP-XG-F are plastically deformed without cracking and have expressed horizontal sections between the two points of inflection. For the systems a transfer from stable condition to plastic flow is estimated by load F^* for $\sim 4.0 \div 5.0$ kPa, a transfer into the condition of the flow with damaged structure occurs at $F^* = \sim 5 \div 7$ kPa. At the same time, rational values plastic yield value K_i , which is determined at the beginning of viscoplastic fluid of disperse system with an undisturbed structure, are within the range of $0.98 \div 1.71$ kPa (Table 3). These values K_i ensure the best extrudability due to their sufficient plasticity and capacity for a viscoplastic flow without the damage of the structure under the influence of compression stress.

The mixes can be extruded through an extruder nozzle with a low squeezing force. The structure of system C2-LF-P-M-F was damaged within the "perfect plastic response phase" (See photo on Fig. 1, a). As a result, this system has not expressed a horizontal section of plastic deformation between the two points of inflection, its values $K_i = 2.20$ kPa. With values, $K_i > 2$ kPa cement-based materials would not possess sufficient extrudability due to insufficient plasticity.

Fig. 1. a) Tested 3D-printable decorative mixtures $F^*(h/R)$ curves;
 b) Tested 3D-printable decorative mixtures "displacement Δ — time t " experimental results;
 c) Tested 3D-printable decorative mixtures "compression force N — displacement Δ " experimental results

At the same time, the structural strength σ_0 is calculated based on the quantity load N at the beginning of deformation of viscoplastic mixes can be considered as the main criterion of their shape stability during loading (Table 3).

Table 3

Rheological properties of 3D-printable decorative mixtures

Specimen ID	Plastic yield value K_i (kPa)	Strength(kPa)		Value of plastic deformations Δ_{pl} (mm/mm)
		Structural σ_0	Plastic σ_{pl}	
C1-LF-P-M-F	0.98	4.69	31.31	0.03
C1-LF-P-PP-XG-F	1.65	1.53	41.66	0.05
C2-LF-P-M-F	2.20	2.19	46.83	0.06
C2-LF-P-PP-XG-F	1.61	0.41	27.10	0.07

The analysis of the obtained experimental data of the curves “displacement Δ – time τ ” (Fig. 1, b) shows that 3 typical sections can be distinguished. The first section is characterized by the lack of deformations under the influence of a load. The quantity of structural strength σ_0 calculated based on the quantity load N at the start of deformation can be considered as the main criterion of shape stability (Table 3). This is a condition due to the fact that structural strength σ_0 characterizes the ability of the system to maintain shape and resist deformation while loaded. The second section is a section of plastic deformation. The ability of the system to deform without destruction is evaluated by the quantity of plastic strength σ_{pl} calculated based on the quantity load N at the beginning of cracking. To characterize the technical efficiency, it seems reasonable to evaluate the number of plastic deformations on this section Δ_{pl} , which have to be minimized for 3D-printable materials. The third section is a section of crack formation and irreversibly destruction of the structure. On the experimental curves “compression force N — displacement Δ ” the moment of the beginning of crack formation is fixed by the peak of the sudden drop of load (Fig. 1, c).

The transition between plastic yield value K_i , the value of structural σ_0 and plastic σ_{pl} strength, plastic deformations Δ_{pl} is linked to the kinds of viscosity modifying additive and cement.

The analysis of the curves “ $\Delta - \tau$ ” and “ $N - \Delta$ ” (Fig. 1, b, c) shows that system C1-LF-P-PP-XG-F and C2-LF-P-PP-XG-F (with xanthan gum + $K_4P_2O_7$) display the longest plastic phase zone. Interval of plastic phase zone for system C1-LF-P-M-F and C2-LF-P-M-F (with metakaolin) decreases by ~2–3 times comparing with the systems modified with xanthan gum + $K_4P_2O_7$. At the same time, the mixture C2-LF-P-M-F has the plastic highest yield value $K_i = 2.2$ kPa (Table 3). This effect of changing the plasticity of 3D-printable mixtures is logically related to the influence of viscosity modifiers. The introduction of different kinds of viscosity modifiers into the concrete mixture as a disperse system is a regulating factor of liquid phase properties. The electrolytes ($K_4P_2O_7$) change the ionic strength of the solution and the energy situation in the condensation volume of the dispersed system. The introduction of inorganic polymers (xanthan gum) changes the density and viscosity of the mixture liquid phase due to the creation of a structural network in its volume. As a result of the complex in-

fluence of xanthan gum + $K_4P_2O_7$, the plasticity of the mixture C1-LF-P-PP-XG-F and C2-LF-P-PP-XG-F increases, while shape stability decreases. Metakaolin as a dispersed adsorption-active inorganic modifier changes the geometric and energetic conditions in the disperse volume of the system. Due to the increase in the viscosity and density of the structured system the shape stability of mixes C1-LF-P-M-F and C2-LF-P-M-F increases.

The kind of cement is a regulating factor of solid-phase properties that changes packing density and molecular interactions between solid particles. The particle size of CEM I 52.5 R is smaller than the particle size of CEM I 42.5 R (Table 2), so the solid phase of the mix with CEM I 52.5 R has less dense packing. Such structuring of the solid phase of the cement mixes based on CEM I 42.5 R ensures the increase of their plasticity and shape stability.

In case of using the same viscosity modifier, the systems based on CEM I 42.5 R have the higher values of structural strength σ_0 and the minimum values of plastic deformations Δ_{pl} in compressing with the systems based on CEM I 52.5 R. According to the experimental data, system C2-LF-P-PP-XG-F does not have the structural stability ($\sigma_0 < 0.5$ kPa).

The heat release during setting and hardening of 3D-printable cement materials depends on the type of cement and viscosity modifiers. The heat release are recorded for mixes C2-LF-P-PP-XG-F and C2-LF-P-M-F based on high-strength Portland cement CEM I 52.5 R (Fig. 2).

Fig. 2. Temperature effects of the 3d-printable fresh mixture during setting

During setting and hardening the temperature of system C2-LF-P-M-F rose to $t \sim 70$ °C, the temperature of system C2-LF-P-PP-XG-F rose to $t \sim 43$ °C. The combination of a highly active aluminosilicate modifier (metakaolin) with high-strength Portland cement CEM I 52.5 R

can cause technologically unacceptable warming of mixes. As a result, heat release in thin 3D-printed structures might provoke significant thermal stresses, deformations, and cracking.

Conclusion. For types of 3D-printable decorative mixes have been obtained using white and grey Portland cement with different particle sizes. It has been established that kind of cement as a factor changing the properties of solid phase in the 3D-printable mixture is an effective method for plasticity regulation and increasing its resistance to load during printing. The shape stability of a 3D-printable mixture can be changed by 2—3 times by means of using cement with efficient ranging of particle size. The 3D-printable mix with CEM I 42.5 R (grey) having ~ 70 % particle with 30—50 μm is characterized by the optimal plasticity, structural strength, and deformability under the load. Using CEM I 52.5 R (white) having ~ 45 % particle with 30—50 μm impairs these characteristics of mixes.

The fresh mixes of 3D-printable decorative concrete had the plastic yield value $K_i \cong 1.0\text{—}2.2$ kPa which is defined the ability of these mixes to plastically deform without structure destruction and maintain their stability during extrusion. At the same time, all the systems with grey Portland cement had a sufficient shape stability (structural strength $\sigma_0 = 1.5\text{—}4.5$ kPa, the value of plastic deformations $\Delta_{pl} = 0.03\text{—}0.05$ mm/mm) which is characterized by the ability of the system to retain its form, resist the deformation under compressions load during multi-layer casting.

The addition of viscosity modifiers with various compositions raises the plastic strength of the mixes by three to five times. It is possible if silicate and aluminosilicate modifier (metakaolin) is used. The reason is that they are similar in their crystal-chemical structure to hydrate phases in the cement systems. The effect of the gum + $\text{K}_4\text{P}_2\text{O}_7$ combined modifier is determined by its impact on the dispersion medium of the cement system. The role of gum is to increase its density and viscosity, while reducing the hydration activity of water molecules. The role of $\text{K}_4\text{P}_2\text{O}_7$ as a strong electrolyte is to change the ionic strength of the solution and raise its supersaturation. As a result, the addition of this modifier allows one to effectively control only the rheological characteristics of the cement systems.

The optimal rheological characteristics of fresh mixes with CEM I 52.5 R (white) can be achieved by means of using metakaolin as a dispersed adsorption-active inorganic modifier changes the geometric and energetic conditions in the volume of the disperse system.

References

1. Slavcheva G., Artamonova O. Reologicheskoe povedenie dispersnykh sistem dlya stroitel'noi 3D-pechati: problema upravleniya i vozmozhnosti arsenala «nano» [The rheological behavior of disperse systems for 3d printing in construction: the problem of control and possibility of «nano» tools application]. Nanotekhnologii v stroi-tel'stve: nauchnyi internet-zhurnal, 2018, no. 10, pp. 107—122.

2. Ur'ev N. B. Fiziko-khimicheskaya dinamika strukturirovannykh nanodispersnykh sistem i nanodispersnykh kompozitsionnykh materialov. Chast' 1 [Physico-chemical dynamics of structured nanodispersed systems and nanodispersed composite materials. Part 1]. Fizikokhimiya poverkhnosti i zashchita materialov, 2010, no. 46, pp. 3—23.
3. Benini H. Concreto Arquitetônico e Decorativo em Concreto. Ensino, Pesquisa E Reliações, IBRACON, 2007, pp. 1413—1551.
4. Benítez A., Bálsamo H. Hormigones arquitectónicos: blancos y coloreados. Hormigones Especiales, AATH, 2004, pp. 75—90.
5. De Schutter G., Lesage K., Mechtcherine V., Nerella V. N., Habert G. & Agusti-Juan I. Vision of 3D printing with concrete-technical, economic and environmental potentials. Cement and Concrete Research, 2018, vol. 112, pp. 25—36.
6. Fernández Gómez J. Estructuras de concreto aparente. Simposio Internacional sobre Concretos Especiais, 2000, pp. 1—22.
7. Figueiredo S. C., Çopuroğlu O., Schlangen E. Effect of viscosity modifier admixture on Portland cement paste hydration and microstructure. Construction and Building Materials, 2019, vol. 212, pp. 818—840. doi: 10.1016/j.conbuildmat.2019.04.020
8. Hou S., Duan Z., Xiao J., Ye J. A review of 3D printed concrete: Performance requirements, testing measurements and mix design. Construction and Building Materials, 2021, vol. 273, pp. 121745. doi: 10.1016/j.conbuildmat.2020.121745
9. Jayathilakage R., Rajeev P., Sanjayan J. G. Yield stress criteria to assess the buildability of 3D concrete printing. Construction and Building Materials, 2020, vol. 240, pp. 117989. doi: 10.1016/j.conbuildmat.2019.117989
10. Khan M. A. Mix suitable for concrete 3D printing: A review. Materials today: Proceedings, 2020, vol. 32 (4), pp. 831—837. doi: 10.1016/j.matpr.2020.03.825
11. Labonnote N., Ronnquist A., Manum B., Rüther P. Additive construction: State-of-the-art, challenges and opportunities. Autom. Constr, 2016, vol. 72, pp. 347—366.
12. López A., Guzmán G. A., Di Sarli A. R. Color stability in mortars and concretes. Part 1: Study on architectural mortars. Constr. Build. Mater, 2016, vol. 120, pp. 617—622. doi: 10.1016/j.conbuildmat.2016.05.133.
13. López A., Guzmán G. A., Di Sarli A. R. Color stability in mortars and concretes. Part 2: Study on architectural concretes. Constr. Build. Mater, 2016, vol. 123, pp. 248—253. doi: 10.1016/j.conbuildmat.2016.06.147.
14. Ma G. W., Wang L. A critical review of preparation design and workability measurement of concrete material for largescale 3D printing. Front Struct Civil Eng, 2018, vol. 12 (3), pp. 382—400. doi: 10.1007/s11709-017-0430-x.
15. Marchon D., Kawashima S., Bessaies-Bey H., Mantellato S., Ng S. Hydration and rheology control of concrete for digital fabrication: Potential admixtures and cement chemistry. Cement and Concrete Research, 2018, vol. 112, pp. 96—110. doi: 10.1016/j.cemconres.2018.05.014.
16. Soltan D. G., Li V. C. A self-reinforced cementitious composite for building-scale 3D printing. Cem Concr Compos, 2018, vol. 90, pp. 1—13. doi: 10.1016/j.cemconcomp.2018.03.017.
17. Souza M. T., Ferreira I. M., Guzi de Moraes E., Senff L., Novaes de Oliveira A. P. 3D printed concrete for large-scale buildings: An overview of rheology, printing parameters, chemical admixtures, reinforcements, and economic and environmental prospects. Journal of Building Engineering, 2020, vol. 32, pp. 101833. doi: 10.1016/j.jobbe.2020.101833.
18. Vergara L. A., Colorado H. A. Additive manufacturing of Portland cement pastes with additions of kaolin, superplasticant and calcium carbonate. Construction and Building Materials, 2020, vol. 248, pp. 118669. doi: 10.1016/j.conbuildmat.2020.118669.
19. Wangler T., Roussel N., Bos F. P., Salet A. M., Flatt R. J. Digital Concrete: A Review. Cement and Concrete Research, 2019, vol. 123, pp. 105780. doi: 105780. 10.1016/j.cemconres.2019.105780.
20. Yu K., Mc Gee W., Ng T. Y., Zhu H., Li V. C. 3D-printable engineered cementitious composites (3DP-ECC): Fresh and hardened properties. Cement and Concrete Research, 2021, vol. 143, pp. 106388. doi: 10.1016/j.cemconres.2021.106388.
21. Yuan Q., Zhou D., Khayat K. H., Feys D., Shi C. On the measurement of evolution of structural build-up of cement paste with time by static yield stress test vs. small amplitude oscillatory shear test. Cem. Concr. Res, 2017, vol. 99, pp. 183—189. doi: 10.1016/j.cemconres.2017.05.014.
22. Zahia T., Roussel N., Lanos C. The squeezing test: a tool to identify firm cement-based material's rheological behaviour and evaluate their extrusion ability. Cem. Concr. Res, 2005, vol. 35, pp. 1891—1899. doi: 10.1016/j.cemconres.2004.09.007.
23. Zhang Y., Zhang Y., Liu G., Yang Y., Wu M., Pang B. Fresh properties of a novel 3D printing concrete ink. Construction and Building Materials, 2018, vol. 174, pp. 263—271. doi: 10.1016/j.conbuildmat.2018.04.115.